CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Industrial Solid Waste

3.1.2 Methanol (Conc 99.9 %)

3.1.3 Ethanol (Conc 99.9 %)

3.1.4 Potassium Sulfate (K_2SO_4)

3.1.5 Potassium Hydrogen Sulfate ($KHSO_3$)

3.2 Equipment

3.2.1 Evaporator (Model: Bushi Rotavapor R-3)

3.2.2 Vacuum Pump (Model: Bushi V-850)

3.2.3 Furnace (Model : YJG 220V 6kW)

3.2.4 Thermalgravimetric analysis (TGA: Model: TA Q-50)

3.2.5 X-Ray Diffraction (XRD: Model: Smartlab)

3.2.6 X-Ray Fluorescence (XRF: Model: AXIOS PW4480)

3.2.7 Atomic Absorption Spectrometer (Model: Varian Spectr AA 300P)

3.3 Methodology

3.3.1 Analysis of Solid Waste Sample

The composition of solid waste would be determined by using TGA.

3.3.1.1 TGA Analysis

The sample was dried in oven at 120 °C for 10 hrs to ensure that water was completely removed from sample. The 10 mg of dried sample was determined decomposing temperature, weight loss and amount of organic compounds by TGA analysis. Furnace was operated at 900 °C with heating rate at 20 °C/min and O_2 flowrate was 20 ml/min. The result was shown as relation between weight of sample and furnace temperature.

3.3.2 Experiment Methods

3.3.2.1 Combustion of Solid Waste Sample

10 g of sample of solid waste was treated in an electrical furnace with heating rate of 6 °C/min to study the effect of temperature. The final temperature was varied from 500 to 600 °C and held at final temperature for 20 mins.

3.3.2.2 Extraction and Precipitation of Solid Waste Sample

10 g of Solid sample was dissolved in 50 ml of water to dissolve inorganic part, and then the insoluble part was filtered out. After that alcohol (such as methanol and ethanol) were used to precipitate inorganic salt out from solution under following condition. This experiment was studied about effect of methanol and ethanol in salt precipitation and effect of alcohol usage. For the effect of methanol and ethanol, volume ratio of water : alcohol was 5:5. And effect of alcohol usage, volume ratio of water : alcohol was varied from 5:1 to 5:7 respectively. The remaining solution from precipitation was evaporated to determine weight of soluble salt. The precipitated salt was dried out water in the oven at 120 °C for 10 hrs and then it was weight to determine sample recovery.

3.3.3 Characterization of Treated Sample

The remaining compounds of waste sample would be determined by using XRD and XRF analysis.

3.3.3.1 Percent Weight Recovery of Treated Sample

Samples were determined weight percent by using Equation 3.1.

$$\%wt.\ Recovery = \frac{W_f}{W_i}x100 \tag{3.1}$$

Where W_i = Sample weight before doing the experiment (g)

W_f = Sample weight after doing the experiment (g)

Precipitated salt was calculated from Equation 3.2.

$$\%Precipitated\ Salt = \frac{W_{precipitated\ salt}}{W_{total\ dissolved\ salt}}x100 \tag{3.2}$$

Total dissolved salt could be calculated from Equation 3.3.

$$W_{total\ dissolved\ salt} = W_{precipitated\ salt} + W_{remaining\ salt} \tag{3.3}$$

Where $W_{precipitated\ salt}$ = Weight of precipitated salt (g)

$W_{remaining\ salt}$ = Weight if salt remaining in aqueous

solution which collected by drying (g)

$W_{total\ dissolved\ salt}$ = Total weight of salt (g)

3.3.3.2 XRF Analysis

In sample preparation method, sample had to mix with boric acid with weight sample to boric acid = 4g : 4g before started analysis. In case of small amount of sample weight sample to boric acid could be 1g : 6g, however the sample had to put in top-center of the sample pan and surround with boric acid to increase accuracy in compound detection.

3.3.3.3 XRD Analysis

The XRD analysis was used to check that the major compound of the sample was K_2SO_4. Scan axis was started from 10 deg to 80 deg in term of Theta/2-theta, with step = 0.01 and speed rate = 3 deg/min.

3.3.3.4 AA Analysis

This method was used to determine compound purity of sample. Sample preparation step was shown in the figure below.

As shown in Figure 3.1, sample 1 and sample 2 was chosen from thermal removal with best result, the result should be pure white powder, and calculated purity of K_2SO_4.
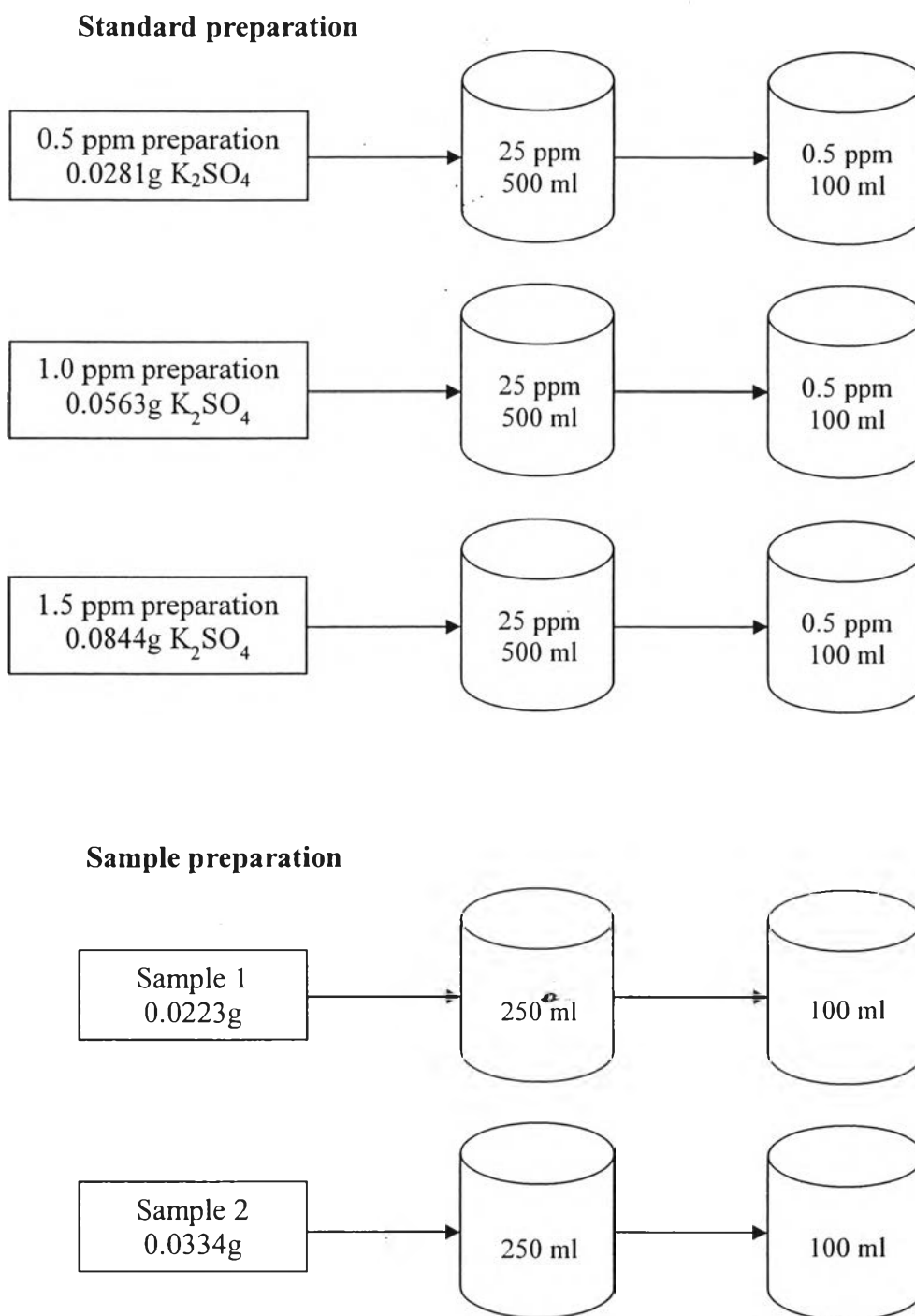
Standard preparation

| 0.5 ppm preparation 0.0281g K$_2$SO$_4$ | → | 25 ppm 500 ml | → | 0.5 ppm 100 ml |

| 1.0 ppm preparation 0.0563g K$_2$SO$_4$ | → | 25 ppm 500 ml | → | 0.5 ppm 100 ml |

| 1.5 ppm preparation 0.0844g K$_2$SO$_4$ | → | 25 ppm 500 ml | → | 0.5 ppm 100 ml |

Sample preparation

| Sample 1 0.0223g | → | 250 ml | → | 100 ml |

| Sample 2 0.0334g | → | 250 ml | → | 100 ml |

Figure 3.1 Standard and sample preparation for atomic adsorption analysis.

3.3.4 Process Flow Diagram

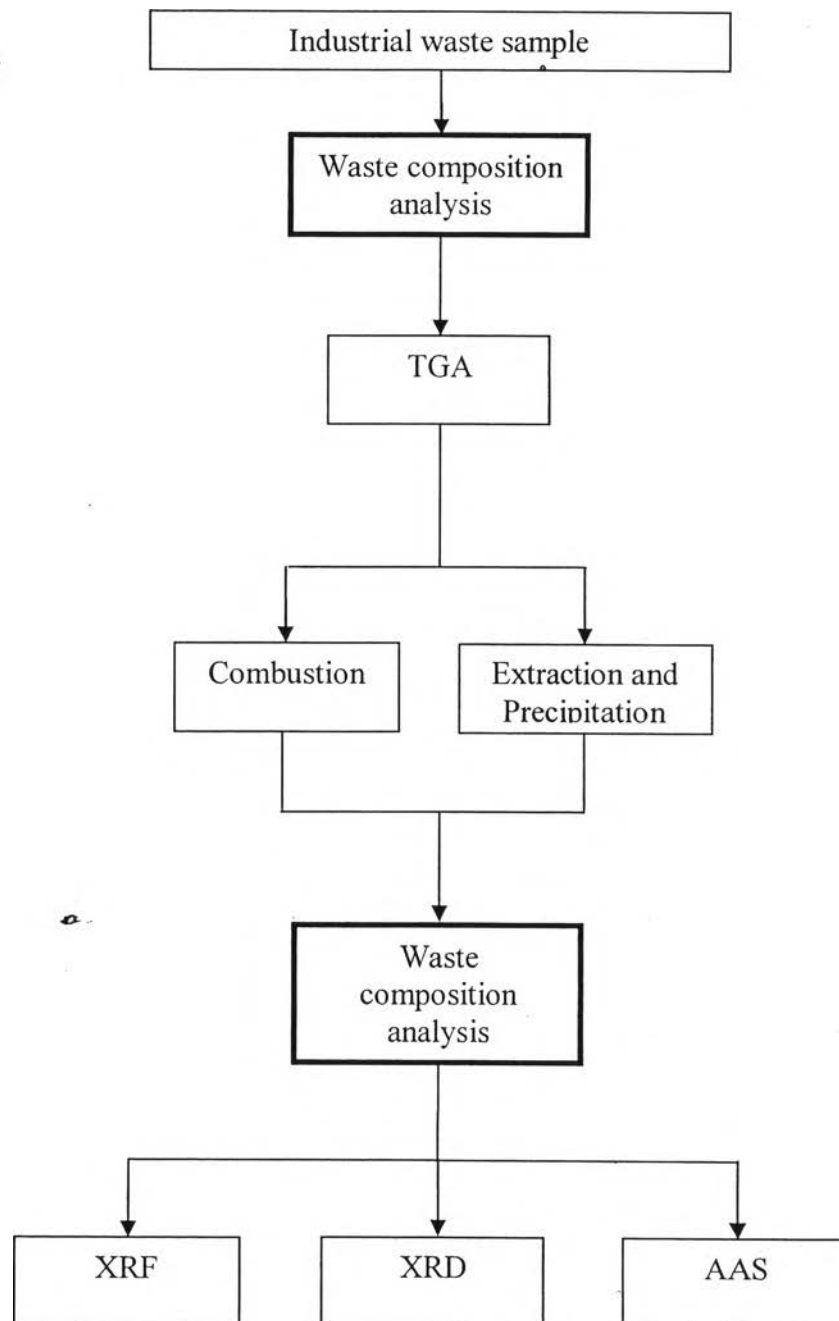Process flow diagram of this experiments is shown in Figure 3.2.

Figure 3.2 Process flow diagram of experiment.

3.3.5 Economic Evaluation

The economic evaluation is basis that production rate = 2,250,000 kg/yr, working time (hr) = 15 hr/day, working time (day) = 25 day/month and the water is totally removed from the biodiesel solid waste.

The predesign estimates are based partially on approximate percentages or factors that are applicable to the process (Peters *et al.*, 2013). For total capital investment cost, the equipment costs are estimated to determine the other costs. Table 3.1 shows the assumed data for each expense in total capital investment at minimum conditions.

Table 3.1 Assumed expenses in total capital investment

Fixed Capital Investment	Assume (%)
Purchased equipment	40 % of Fixed capital investment
Purchased equipment installation	6 % of Fixed capital investment
Instrument and control (installed)	2 % of Fixed capital investment
Valve and Piping (installed)	3 % of Fixed capital investment
Electrical (installed)	2 % of Fixed capital investment
Building (include services)	3 % of Fixed capital investment
Land	Land cost is referred from Department of Land (Rayong, Sukhumwit Road)
Yard improvements	2 % of Fixed capital investment
Service facilities (installed)	8 % of Fixed capital investment
Engineering and supervision	4 % of Fixed capital investment
Construction expense	4 % of Fixed capital investment
Contractor's fee	2 % of Fixed capital investment
Contingency	5 % of Fixed capital investment
Working Capital	10 % of Total capital investment

Table 3.2 shows about total production cost at minimum conditions, the utilities are the main expense to be estimated to determine the other annual operation costs.

Table 3.2 Assumed expenses in total production cost

Direct Production Cost	Assume (%)
Raw material	-
Operating labor	15 % of Total production cost
Direct supervisory and clerical labor	10 % of Operating labor
Utilities	15 % of Total production cost
Maintenance and repairs	2 % of Fixed capital investment
Operating supplies	10 % of Maintenance and repair cost
Laboratory charges	-
Patents and royalties	-
Fixed Charge	
Depreciation	10 % of Fixed capital investment
Local taxes	1 % of Fixed capital investment
Insurance	0.4 % of Fixed capital investment
Rent	-
Plant-overhead	5 % of Total production cost
General Expense	
Administrative costs	2 % of Total production cost
Distribution and selling costs	2 % of Total production cost
Research and development costs	2 % of Total production cost
Financing	-

The total production costs and payback period as well as product price are calculated based on the simulation results. The total production costs are the sum of raw materials and items in operating cost.

Total product price is then calculated by using Equation 3.4 and Equation 3.5.

$$Product\ price = \frac{Total\ production\ cost}{Production\ rate} \qquad (3.4)$$

$$Net\ profit = Product\ sale - Total\ production\ cost \qquad (3.5)$$

The product sales are assumed to be 15, 20, and 25 bath/kg respectively to determine payback period. Then payback period is calculated by using Equation 3.6.

$$Payback\ period = \frac{Capital\ cost}{Net\ profit} \qquad (3.6)$$

Both processes, combustion, and extraction and precipitation are compared to each other from result of payback period.